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(54) **Method of making submicron cemented carbide cutting tool inserts**

(57) The present invention relates to an improved method of making submicron cemented carbide cutting tool inserts consisting of tungsten carbide and cobalt by the conventional methods wet milling of powders of WC and Co and conventional grain growth inhibitors to a slurry, drying said slurry to a powder, uniaxial pressing in pressing tools of the powder to bodies of desired shape and finally sintering. During sintering, inserts of this type of cemented carbide generally shrink more in the direction parallel to the pressing direction than in the

direction perpendicular thereto. As a consequence, the pressing has to be done in special tools or the inserts have to be extensively ground after sintering both alternatives leading to increased production cost. According to the invention it has been found that for a cemented carbide with a Co-content of 7.5-25 wt% this disadvantage can be eliminated by using WC powder with an FSSS grain size,  $d_{WC}$ , of less than 1  $\mu m$  and a Co powder with an FSSS grain size,  $d_{Co}$ , such that the ratio  $d_{WC}/d_{Co}$  is 0.75-1.5.

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## Description

[0001] The present invention relates to an improved method of making submicron cemented carbide cutting tool inserts consisting of tungsten carbide and cobalt.

[0002] Manufacture of cemented carbides involves wet milling of powders forming a binder phase and hard constituents, drying the slurry to a powder, uniaxial pressing in pressing tools of the powder to bodies of desired shape and finally sintering. During sintering, the bodies shrink approximately 17-18% linearly. In general, the shrinkage is essentially isotropic both parallel to and perpendicular to the pressing direction. However, for submicron grades (i.e. in which essentially all of the WC grains are less than 1  $\mu\text{m}$ ) the shrinkage is anisotropic. The shrinkage parallel to the pressing direction is larger than that perpendicular thereto.

[0003] One way to define the character of the shrinkage is by means of the K-value according to:

$$K = \frac{h_s \cdot w_p}{h_p \cdot w_s}$$

where

$h_s$  = the sintered height

$w_p$  = the pressed width

$h_p$  = the pressed height

$w_s$  = the sintered width

[0004] The height is defined as the dimension in the pressing direction and the width that perpendicular thereto.

[0005] For a completely isotropical shrinkage  $K=1.000$ , while for a submicron grade K is less than 1.000. For the submicron grades K depends on the cobalt content being close to 1.000 for grades containing about 6 wt% Co down to 0.960 for grades containing 20 wt% Co.

[0006] One conventional way to characterize the average grain size of a powder is by means of the Fisher Sub-Sieve Sizer (FSSS). This apparatus employs the air permeability method in which the pressure drop over a certain amount of powder is registered and converted into an FSSS average grain size value.

[0007] US 5,441,693 discloses in Examples 1 and 2 the use of 0.4  $\mu\text{m}$  Co-powder in a submicron WC with 6.5 and 6 wt% Co, respectively.

[0008] In JP 51-126 309 the manufacture of cemented carbide with a WC grain size of 0.5-0.8  $\mu\text{m}$  and 12 wt% Co with a grain size of 1  $\mu\text{m}$  is disclosed.

[0009] EP-A-0 380 096 discloses in Example 3 the manufacture of a drill shank portion by mixing WC 0.8  $\mu\text{m}$  and Co 0.5  $\mu\text{m}$  in a relative amount of 15 to 23 vol% corresponding to about 9.5 to 14.5 wt% Co.

[0010] As already mentioned the shrinkage is anisotropic for submicron cemented carbide grades. This means that special pressing tools have to be made for

pressing of the submicron grades, which is a large disadvantage since pressing tools are expensive to produce. Alternatively, the sintered bodies have to be subjected to an extensive grinding operation which is expensive and time consuming.

[0011] It is therefore an object of the present invention to provide a method of avoiding special pressing tools or post-sintering grinding for the manufacture of submicron cemented carbides.

[0012] According to the present invention it has now surprisingly been found that the use of a cobalt powder with essentially the same grain size as the WC-powder results in a K-value approximately equal to 1.000.

[0013] More particular the present invention relates to a submicron cemented carbide cutting tool in which essentially all of the WC grains are less than 1  $\mu\text{m}$ , preferably 0.2 to 0.9  $\mu\text{m}$  and with a cobalt content of 7.5 to 25 wt%, preferably 9 to 20 wt%, most preferably 10 to 15 wt%. In addition, the material contains conventional grain growth inhibitors such as carbides of tantalum, chromium and/or vanadium generally up to 1 wt%, in the case of tantalum carbide only, up to 1.5 wt%.

[0014] According to the method of the present invention a submicron cemented carbide cutting tool is manufactured by wet milling a slurry consisting of WC powder with an FSSS grain size,  $d_{WC}$ , of less than 1  $\mu\text{m}$  and preferably 0.1 to 0.9  $\mu\text{m}$  and most preferably 0.2 to 0.8  $\mu\text{m}$  and Co powder in above mentioned amounts preferably with an FSSS grain size,  $d_{Co}$ , of less than 1  $\mu\text{m}$  such that the ratio  $d_{WC}/d_{Co}$  is  $>0.75$ , preferably  $>0.85$ , most preferably  $>0.90$  and  $<1.5$ , preferably  $<1.3$ , most preferably  $<1.2$ . It is essential that the FSSS-value is determined on deagglomerated powders, since determination on agglomerated powders gives incorrect results. In addition, conventional grain growth inhibitors are added in above-mentioned amounts together with usual pressing aid.

[0015] The obtained slurry is dried to a powder with good flowability. This powder is uniaxially pressed in a pressing tool to a body of desired shape. Then, this body is sintered to a cutting tool insert. The pressing tool is the same as that used for making cemented carbides with medium to coarse WC grain size. The sintered insert does not require any further grinding other than that generally necessary for corresponding medium to coarse grained grades.

### Example 1 (prior art)

[0016] A WC-10 wt% Co submicron cemented carbide was made by wet milling 300 g Co-powder (Westaim 2M) with an FSSS average grain size of 1.81  $\mu\text{m}$ , 14.85 g  $\text{Cr}_3\text{C}_2$  (H C Starck), 2683.1 g WC (H C Starck) with an FSSS average grain size of 0.83  $\mu\text{m}$ , 2 g carbon black and 75 g PEG in 0.8 l milling liquid consisting of ethylalcohol and water (70:30 by volume) for 40 h. The resulting slurry was spraydried to a powder from which test samples were pressed at 171.6 MPa.

The samples had the dimensions 15.39x15.39x6.51 mm<sup>3</sup>. The latter dimension was parallel to the pressing direction. The samples were sintered at 1410 °C in Ar at a pressure of 4kPa. After sintering the samples had the dimensions 12.75x12.75x5.34 mm<sup>3</sup> resulting in a K-value of 0.990. 5

characterized in that the ratio  $d_{WC}/d_{Co}$  is  $>0.90$  and  $<1.2$ .

#### Example 2

[0017] Example 1 was repeated with a Co-powder with an FSSS average grain size of 0.90 µm (Westaim ultrafine). The pressed test samples had in this case the dimensions 15.39x15.39x6.54 mm<sup>3</sup>. The sintered test samples had the dimensions 12.66x12.66x5.36 mm<sup>3</sup> resulting in a K-value of 0.996. 10 15

#### Example 3 (comparative)

[0018] A WC-20 wt% Co submicron cemented carbide was made in the same way as in Example 1 but with the use of a WC-powder with an FSSS average grain size of 0.4 µm (H C Starck) and a Co-powder with an FSSS average grain size of 2 µm (OMG). A K-value of 0.964 was obtained. 20 25

#### Example 4

[0019] Example 3 was repeated but with a Co-powder with an FSSS average grain size of 0.4 µm (ETP). A K-value of 0.988 was obtained. 30

#### **Claims**

1. Method of making submicron cemented carbide cutting tool inserts consisting of WC and Co by wet milling of powders of WC and Co and conventional grain growth inhibitors to a slurry, drying said slurry to a powder, uniaxial pressing in pressing tools of the powder to bodies of desired shape and finally sintering characterized in that the cemented carbide has a Co-content of 7.5-25 wt% and that the WC powder has an FSSS grain size,  $d_{WC}$ , of less than 1 µm and the Co powder has an FSSS grain size,  $d_{Co}$ , such that the ratio  $d_{WC}/d_{Co}$  is  $>0.75$  and  $<1.5$ . 35 40 45
2. Method according to claim 1 characterized in that the Co-content is 9-20 wt%. 50
3. Method according to claim 1 characterized in that the Co-content is 10-15 wt%.
4. Method according to any of the preceding claims characterized in that the ratio  $d_{WC}/d_{Co}$  is  $>0.85$  and  $<1.3$ . 55
5. Method according to any of the preceding claims



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Application Number  
EP 99 85 0024

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			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 15 June 1999	Examiner Schruers, H
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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**ANNEX TO THE EUROPEAN SEARCH REPORT  
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on  
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